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INFLUENCE OF CRUCIBLE MATERIALS ON HIGH-TEMPERATURE PROPERTIES OF VACUUM-MELTED NICKEL-CHROMIUM-COBALT ALLOY

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INFLUENCE OF CRUCIBLE MATERIALS
ON HIGH-TEMPERATURE PROPERTIES OF VACUUM-MELTED
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SUMMARY

A study of the effect of induction-vacuum-melting practice on the high-temperature properties of 55Ni-20Cr-15Co-4Mo-3Ti-3Al alloy revealed that a major variable was the type of ceramic used as a crucible. Reactions between the melt and magnesia or zirconia crucibles apparently increased high-temperature properties at 1,600° F by introducing small amounts of boron or zirconium into the melts.

Heats melted in alumina crucibles had relatively low rupture life and ductility at 1,600° F and were prone to crack during hot-working. Apparently this resulted from lack of derivation of boron or zirconium from the crucible.

When melting was carried out in zirconia crucibles with a variety of melting practices, increases in rupture life and ductility and reduction of cracking during hot-working correlated with increases in zirconium content derived from the crucibles. However, the best heats made in zirconia crucibles had properties below those considered characteristic of the alloy.

Melting in magnesia led to heats with rupture properties and resistance to cracking during hot-working which were improved over those of the heats made in alumina. The rupture life was increased to the range considered characteristic of the alloy. This appears to be related to boron content resulting from crucible reaction.

Controlled additions of boron and zirconium made to heats melted in alumina crucibles gave heats with properties which correlated with the earlier results from heats where zirconium was derived from zirconia and boron from magnesia. This supports the postulation that the introduction of these elements through melt reaction with the crucibles was responsible for the improved properties.

Simultaneous additions of boron and zirconium to experimental heats gave higher creep-rupture properties than additions of either element alone. There appear, however, to be sharp optimum amounts or combinations of the two elements from the standpoint of both strength and cracking during hot-working. Too much boron and zirconium together can result in rather low properties.

These effects appear to account for many of the unexplainable variations in properties of the alloy studied, as well as for those of other nickel-base titanium-and-aluminum-bearing alloys. In particular, variable boron contents derived from the magnesia melting-furnace linings used commercially or introduced unknowingly with the charge seem to be important.

INTRODUCTION

A relationship between the type of refractories used in the induction vacuum melting of an alloy and its properties at high temperatures was established during the course of an experimental program on the influence of melting-practice variables on the high-temperature properties of a 55Ni-20Cr-15Co-4Mo-3Ti-3Al alloy.

Initially, the major objective was to establish the role of oxygen and nitrogen content. Induction vacuum melting in zirconia crucibles was adopted for initial phases of the investigation. It soon became evident that the strengths at 1,600° F of the heats being made were below those considered characteristic of the alloy. Numerous modifications of melting and casting procedures were carried out in an effort to produce properties in the experimental heats comparable with those in the commercial heats. These modifications were investigated in view of positive statements that no special additions were made commercially and that the cause of the low strength must be in the particular procedure used.

Considerable variation in properties was obtained. However, the best heats were still on the low-strength side of the strength-range characteristic of the alloy. The development of adequate methods of analysis for zirconium by the Utica Drop Forge and Tool Corp. and its cooperation in making analyses finally disclosed that the properties correlated with zirconium content derived from reaction between the melt and the crucibles. No significant effect was ever identified for the oxygen or nitrogen contents or for the other melting-practice variables studied.

Deliberate boron additions to the heats produced material with properties equal to or superior to those considered characteristic of the alloy. It was concluded that this was the only way to produce high

strength and ductility in the alloy. Simultaneously a comprehensive report (ref. 1) was made available through the Universal-Cyclops Steel Corp. showing systematically the improvements in properties from boron and zirconium additions to the alloy. In view of the undoubted fact that high levels of good properties were being produced commercially without intentional additions of either boron or zirconium, it was decided to experiment with other refractories for crucibles. Accordingly, heats were made in both alumina and magnesia crucibles. The properties of the material melted in alumina were inferior to those of the material melted in zirconia. The heats made in magnesia, the refractory most used commercially, were superior in rupture life to those made in zirconia and comparable with commercially produced material.

In view of the disclosures of the Universal-Cyclops Steel Corp. of the effects of boron and zirconium, its assistance in analyzing for boron was obtained. Very little boron was found in the heats made in alumina while the magnesia-made heats contained minute but significant amounts. It was originally thought that the magnesia crucible might have a beneficial effect through introduction of magnesium to the melt. No evidence to support this has been found and the boron pickup appears to be adequate to explain the results.

The literature cites numerous cases where beneficial effects were obtained by minute additions of certain alloying elements (ref. 2). Deoxidation with certain elements, including boron and zirconium, has been reported to improve hot-workability and properties at high temperatures of nickel-base alloys (refs. 3 to 5). Variation in properties of alloys at high temperatures from variations in melting and deoxidation practice has long been known. Unidentified heat-to-heat variations with supposedly constant melting practice frequently occur. The present report discloses that one cause of such effects apparently arises from variable minute additions of certain elements through reaction between the melt and refractories.

The disclosure of the role of zirconium and boron as the key to the influence of the type of crucible ceramic was due to the cooperation and analytical skill of the chemists at the Utica Drop Forge and Tool Corp. and Universal-Cyclops Steel Corp. This part of the research was in fact made possible because their skill made methods of analysis available as the need arose.

The invaluable contribution of a number of people who have assisted on this project is acknowledged. Professor Lars Thomassen's enlightening advice on possible effects of zirconium derived from the zirconia crucibles was very helpful. Mr. Karl Kienholz operated the University of Michigan vacuum-melting furnace in the processing of the experimental heats. Mr. Jerry White assisted during hot-working and heat-treated many of the samples. Mr. Alex Dano's development of etching procedures for both

macroscopic and optical-microscopic work and the subsequent utilization of the techniques by Mr. Dano aided greatly in the study. Miss Christine Sadler has ably prepared metallographic samples and photographs. Mr. George Hynes and Mr. Dick Umstead ran the stress-rupture tests.

The investigation was conducted at the Engineering Research Institute of the University of Michigan under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

EXPERIMENTAL PROCEDURES

Melting

The experimental heats were induction-melted in the University of Michigan vacuum-melting unit shown in figure 1. Pressures before melt down and after pouring were less than 5 microns as measured by both Stokes and thermocouple gages. No gases were purposely added to the chamber during the melting cycle. Melt temperatures were measured with an immersion thermocouple with one platinum and one platinum and rhodium wire. Melting cycles for all the heats are detailed in table I and figure 2.

The analysis aimed at for the basic alloy, in weight percent, for all heats was as follows:

Chromi- um, Cr	Cobalt, Co	Molyb- denum, Mo	Titani- um, Ti	Alumi- num, Al	Manga- nese, Mn	Silicon, Si	Car- bon, C	Nickel, Ni
20.0	15.0	4.0	3.1	3.1	0.12	0.12	0.08 to 0.15	Balance

Varied amounts of boron and zirconium were added to certain heats for the purposes of the investigation.

Laboratory-grade electrolytic nickel, chromium, cobalt, and manganese melting stocks were used. The molybdenum was arc-melted, low-carbon stock; the titanium was Ti-55A stock; the aluminum was 99.99-percent-pure ingot stock; and the silicon was 99.9-percent-pure powder; zirconium was added as sponge, boron, as nickel-boron master alloy, and carbon, as powdered or chunk graphite.

The melting-crucible refractories were alumina, zirconia, and magnesia. Pertinent data on these materials are listed in table II with chemical compositions obtained from the suppliers.

Ten-pound heats were poured into an open-bottomed copper mold which rested on a massive copper block. An insulating refractory ring mounted in the top of the copper mold served as the hot top. An as-cast ingot, a hot-top section, and the ingot structure are shown in figure 3.

Hot-Working

The hot-working practice was kept constant for the majority of the heats as follows:

- (1) Ingot was homogenized 1 hour at 2,300° F, then air-cooled.
- (2) Ingot was surface-ground.
- (3) Ingot was rolled at 2,150° F to 7/8-inch bar stock using 22 passes with 21 reheats of 10 minutes between passes. The last pass was a 7-percent reduction followed by air-cooling.

The deviations from this practice were heats V-20, V-21, and V-24, which were not homogenized or ground, and heats V-18 and V-19, which were rolled at 2,000° F to 7/8-inch bar stock using 36 passes with 35 reheats.

Chemical Analysis

Samples for chemical analysis were cut from the midpoint of the as-rolled bar stock. This metal was originally in the center of the ingot. Titanium, aluminum, molybdenum, chromium, cobalt, manganese, magnesium, copper, iron, and zirconium were determined spectrographically through the courtesy of the Utica Drop Forge and Tool Corp. Carbon, sulfur, and phosphorus were determined by chemical analysis of machined chips. Residual calcium, iron, copper, and magnesium were checked spectrographically on heats V-13, V-20, and V-21. The difficult and time-consuming analyses for boron were provided gratis by the Universal-Cyclops Steel Corp. from chemical determinations.

Metallography

Macrostructures.- The ingot section was prepared for macroetching by mechanical polishing with 3/0 paper. The macroetching was a two-step procedure as follows:

- (1) Etched 5 minutes in the following solution:

<u>Component</u>	<u>Percent by volume</u>
Sulfuric acid (96 percent)	21
Hydrochloric acid (38 percent)	15
Nitric acid (70 percent)	21
Hydrofluoric acid (48 percent)	21
Water	22

- (2) Etched 5 minutes in the following solution:

<u>Component</u>	<u>Percent by volume</u>
Cupric chloride solution (solution of 1 gram $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ per 5 milliliters H_2O)	40
Hydrochloric acid (38 percent)	40
Hydrofluoric acid (48 percent)	20

Microstructures.— The samples were mechanically polished with 3/0 paper and then electropolished in a solution of 10 parts of perchloric acid (70 percent) and 90 parts of glacial acetic acid. Electropolishing was for 30 seconds at approximately 30 volts and 0.8 ampere per square inch. Cyclic polishing of 5 seconds on and 5 seconds off was employed. Then the samples were etched at 4 volts and 0.4 ampere per square inch for 10 seconds using the following etchant:

<u>Component</u>	<u>Percent by volume</u>
Cupric chloride solution (solution of 1 gram $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ per 5 milliliters H_2O)	29
Acetic acid (glacial)	36
Hydrochloric acid (38 percent)	23
Sulfuric acid (96 percent)	5
Chromic acid (solution of 1 gram CrO_3 per 3 milliliters H_2O)	7

Stress-Rupture Testing

The high-temperature properties of the experimental heats were evaluated by stress-rupture testing. The as-rolled bar stock was treated 2 hours at 2,150° F, air-cooled, and machined into 0.250-inch-diameter rupture specimens. The specimens were preheated 4 hours at 1,600° F and loaded at 1,600° F and 25,000 psi.

RESULTS

In this study the lowest rupture life and ductility were found in heats melted in alumina crucibles while heats melted in zirconia and magnesia crucibles had greater rupture life and ductility. Therefore, for purposes of this report, the heats melted in alumina are considered baseline material.

Chemical Composition

The results of the chemical analyses are given in table III. It was not possible to obtain boron analyses on all heats. It should be noted that heats melted in alumina crucibles had less than 0.0004 percent of boron and less than 0.01 percent of zirconium. The zirconium was also less than 0.01 percent in heats melted in magnesia crucibles. The lower limit of the zirconium-analysis sensitivity was 0.03 percent in the earlier heats, heats V-1 through V-10. The sensitivity was improved to 0.01 percent in the later heats, heats V-11 through V-24. The heats melted in zirconia crucibles contained variable amounts of zirconium. Exceptions to these generalities occurred only when boron or zirconium were deliberately added to the heats.

The degree of reliability of analyses for magnesium, calcium, and copper is uncertain. Some variation in aluminum and titanium is indicated by the results of the analyses.

Hot-Working

Working conditions for the alloy were found limited in temperature range, amount of reduction per pass, and the reheat time between passes. This was due to the tendency for cracking to occur. The most successful practice found for the heats with less than 0.01 percent zirconium and less than 0.001 percent boron was to roll from 2,150° F using approximately 7-percent reductions per pass with a 10-minute reheat at 2,150° F between passes. Therefore, this practice was adopted for all rolling except for heats V-18 and V-19, which were rolled from 2,000° F with

smaller reductions because of the hot shortness related to combined content of boron and zirconium. Because of these limitations of hot-working, evaluation of hot-working characteristics has been restricted to observation of cracking during rolling.

Observation of hot-working revealed that two distinctly different types of cracking occurred. The first type was a severe internal rupturing of the stock. The appearance of one of these ruptures is shown in figure 4. The second type of cracking occurred after the round ingot had been broken down to square bar stock in the rolling mill. This was corner cracking, which did not propagate far into the bar stock. Typical corner cracking is shown in figure 5. It was found that both types of cracking were related to trace element content.

Internal cracking during rolling.- The relationships governing internal rupturing during ingot breakdown were:

(1) The base alloy as melted in alumina was not subject to internal rupturing.

(2) Zirconium content of the heats melted in zirconia or alumina without boron additions apparently did not introduce internal rupturing. The relation held up to a zirconium content of 0.19 percent (heat V-6).

(3) Increasing boron content in the presence of less than 0.01 percent zirconium did introduce internal rupturing. This was first noticed in the finished bar stock of heat V-12 (0.0089 percent B) which exhibited some small voids along the center line which resulted from internal rupture during breakdown.

(4) Presence of both boron and zirconium had a pronounced effect. Severe internal rupturing occurred during ingot breakdown. Figure 4 illustrates the severity of this in heat V-14 which contained 0.0088 percent boron and 0.01 percent zirconium. Thus, the small increase in zirconium content from less than 0.01 percent (the limit of spectrographic analysis) in heat V-12 to a definite 0.01 percent in heat V-14 in the presence of a boron content of near 0.009 percent was enough to magnify internal rupturing. In general, the severity of the internal rupturing in the heats containing 0.0069 to 0.0090 percent boron (heats V-12, V-14, V-17, V-18, and V-19) increased with zirconium content in the range covered from less than 0.01 to 0.09 percent zirconium. Heats V-18 (0.0069 percent B and 0.09 percent Zr) and V-19 (0.0090 percent B and 0.04 percent Zr) were very hot short during the first passes in the standard rolling schedule. The rolling temperature had to be dropped to 2,000° F and the reduction per pass, to 3 percent in order to minimize internal rupturing during ingot breakdown and during subsequent rolling of the recrystallized bar stock.

(5) The time of internal rupturing in the rolling cycle was a function of the boron and zirconium levels. In heat V-14 (0.0088 percent B and 0.01 percent Zr), internal rupturing was limited to the first three passes at 2,150° F where it occurred along the as-cast grain boundaries. Once the original as-cast structure was recrystallized, internal rupturing ceased and the stock was reduced to 7/8-inch bar stock successfully with the standard rolling procedure. However, in heat V-18 (0.0069 percent B and 0.09 percent Zr) and heat V-19 (0.0090 percent B and 0.04 percent Zr), internal rupturing persisted after recrystallization of the as-cast grains. In order to minimize this rupturing throughout the rolling cycle, the rolling temperature was lowered to 2,000° F and the reductions per pass to 3 percent. With this procedure, it was possible to obtain some useful stock for rupture testing.

The 2,300° F treatment included in the standard rolling practices was first adopted for heats with no boron addition in the belief that it would minimize corner-cracking problems during rolling by homogenizing the as-cast ingot. It is possible that this treatment magnified the internal-rupture problem in heats to which boron was added. The introduction of the 2,300° F treatment was based on experience with one of the higher zirconium heats and it is now considered that the zirconium and not the heat-treatment was responsible for the improved hot-workability.

Corner cracking during rolling.- After the round ingots had been broken down to $1\frac{3}{16}$ -inch squares in the rolling mill, shallow corner cracking occurred in some of the experimental heats. These cracks could be ground out between passes rather easily but reoccurred during subsequent passes. The severity of this cracking was variable; some of the heats were very malleable with no corner cracking while others underwent cracking with each pass.

As seen in figure 5, the relationships governing corner cracking during rolling of the bar stock were:

(1) In heats melted in alumina or zirconia crucibles with no boron additions, cracking decreased with increasing zirconium content.

(2) In heats with less than 0.01 percent zirconium (melted in alumina or magnesia crucibles), cracking decreased with increasing boron content.

(3) Little corner cracking occurred in the heats containing moderate amounts of both boron and zirconium.

Stress-Rupture Properties

Stress-rupture data for tests at 1,600° F and 25,000 psi on the experimental heats are listed in table IV.

Major effects related to the zirconium content of the heats were found. In the heats melted in zirconia with about 0.0005 percent boron, as the zirconium content derived from the crucibles increased from less than 0.01 to 0.19 percent, the average rupture life increased from 50 to 140 hours, the average elongation of heats with less than 0.10 percent carbon increased from 2 to 6 percent, and the average reduction of area of heats with less than 0.10 percent carbon increased from 1 to 6 percent.

In addition, major effects related to the boron content of the heats were established. In the heats melted in alumina or magnesia crucibles with less than 0.01 percent zirconium, as the boron content increased from 0.0002 to 0.0089 percent the average rupture life increased from about 50 to 411 hours, the average elongation increased from 2 to 8 percent, and the average reduction of area increased from 1 to about 10 percent.

Simultaneous additions of both boron and zirconium were found to have even larger effects.

The general range of the effects on rupture strength is shown by figure 6 which compares the range of rupture times with two curves stated to be characteristic of commercially produced material. The effects of the various compositional variations on creep characteristics are demonstrated by figure 7. The properties at 1,600° F are presented in detail in the following section of the report. --

Effect of zirconium and boron derived from crucibles.— Heats V-1 to V-11, V-13, V-15, V-20, and V-21 were melted in order to study the effect of vacuum-melting practice on the high-temperature properties of the alloy. Among these heats, a range of rupture life from 44.6 to 229.9 hours was obtained while the elongation varied from 1 to 11 percent and the reduction of area ranged from 1 to 12 percent.

Analysis of these ranges of rupture life and ductility revealed that a close relationship existed among crucible materials, resulting content of zirconium and boron, and the rupture properties. This relationship held despite the multiplicity of melting variables, including refining time and temperature, deoxidation material, superheat temperature, pouring temperature, crucible material, meltdown time, and ingot-mold shape.

Heats V-1 to V-10, melted in zirconia crucibles with no zirconium additions, had varied zirconium contents. These contents ranged from less than 0.03 percent, the limit of analysis in the early heats, to

0.19 percent. Correlation with the rupture life (fig. 8) shows the regular increase in strength with zirconium content. The rupture life at a given zirconium content seemed to be dependent on the titanium and aluminum content of the alloy. This is indicated in figure 8 by the arbitrary grouping of the heats as greater than or less than 6.50 percent titanium plus aluminum. The purpose was to illustrate that, for a given zirconium content, rupture life appeared to be related to titanium plus aluminum content. The ductility of the heats (fig. 9) also increased with zirconium content with the exception of heats V-8, V-9, and V-10, in which higher carbon content (0.13 to 0.20 percent C) evidently increased ductility.

Heats V-11, V-20, and V-21, which were melted in magnesia crucibles, had higher boron content than comparable heats made in alumina crucibles. Rupture life and ductility of these heats are plotted in figures 10 and 11 to show the increase of rupture properties with boron content.

The heats melted in an alumina crucible, V-13 and V-15, had comparatively low boron content (0.0003 percent or less) and low zirconium content (less than 0.01 percent). Corresponding low rupture life (44.6 to 56.9 hours) and ductilities (1 to 2 percent) were exhibited by these heats. These values are included in the correlations of figures 8 to 11.

From the rupture data on heats melted in zirconia, magnesia, and alumina crucibles without deliberate additions of boron and zirconium, it is apparent that melt reactions with crucible materials can be an important melting variable. The zirconium content of 0.19 percent in heat V-6 accounted for an increase in average rupture life from 50 to 140 hours, an increase in average elongation from 2 to 6 percent, and an increase in average reduction of area from 1 to 6 percent over heats V-13 and V-15 melted in alumina crucibles. Likewise, the portion of the 0.0010 percent boron of heat V-20 derived from the magnesia crucible accounted for an increase of average rupture life from 50 to 216 hours, an increase in average elongation from 2 to 4 percent, and an increase in average reduction of area from 1 to 4 percent over heats V-13 (0.0003 percent B) and V-15 (0.0002 percent B).

The correlations of rupture properties with zirconium and boron contents derived from reaction with crucible materials apparently account for a major portion of the variability of rupture properties with vacuum-melting practice employed in this work. From the results, it can be observed that derivation of boron from magnesia can be a more potent variable in rupture life than derivation of zirconium from zirconia. However, the effects on ductility are about equal.

Much of the scatter of data points from the curves of correlation resulted from normal within-heat scatter expected in chemical testing at trace levels and from preparation and testing of rupture specimens.

It is possible that deviations were caused by the other variables investigated: Refining practice, deoxidation practice, superheat temperature, pouring temperature, melt-down time, and ingot-mold shape. However, it can be seen that these variables were minor in comparison with the effects of boron and zirconium as far as the limited scope of this study is concerned.

Effect of zirconium additions.- Recovery of 13 percent zirconium from a deliberate addition of zirconium to heat V-16, which was melted in an alumina crucible, resulted in an average rupture life of 131 hours and average elongation and reduction of area of 4 percent. The rupture life agreed with the correlation developed from the heats in which zirconium was derived from zirconia crucibles (fig. 8).

Although close agreement of the ductility of this heat with the correlation of figure 9 was not obtained, an increase in ductility from 1.5 to 4 percent was attributable to the zirconium content. Thus, the results suggest that the crucible effect arises mainly from the final zirconium content of the heat and from the particular crucible material used only as it affects final zirconium content.

Effect of boron additions.- Recovery of 0.0012 percent boron from a deliberate addition of boron to a heat melted in an alumina crucible (heat V-24) increased rupture life to 170 hours, elongation to 5 percent, and reduction of area to 3 percent. These values were increased over those of the baseline heats V-13 (0.0003 percent B) and V-15 (0.0002 percent B) which had an average rupture life of 50 hours, an average elongation of 2 percent, and an average reduction of area of 1 percent.

A boron recovery of 0.0074 percent from a deliberate boron addition to a heat melted in an alumina crucible (heat V-17) caused a marked increase of average rupture life (50 to 373 hours), an increase in average elongation (2 to 7 percent), and an increase in average reduction of area (1 to 6 percent) over heats V-13 (0.0003 percent B) and V-15 (0.0002 percent B). A boron recovery of 0.0089 percent from a boron addition to a heat melted in a magnesia crucible (heat V-12) caused an increase in average rupture life from 50 to 411 hours, in average elongation from 2 to 8 percent, and in average reduction of area from 1 to 10 percent over the heats melted in alumina crucibles (heats V-13 with 0.0003 percent B and V-15 with 0.0002 percent B).

The results are included in figures 10 and 11. It is apparent that these data supplement those on derivation of boron from magnesia and seem to substantiate that effect by extending the correlation to higher boron contents.

The combination of a recovered addition of 0.0089 percent boron with a zirconia crucible (heat V-14) caused the even greater increase in average rupture life of 50 to 646 hours, in average elongation from 2 to 14 percent, and in average reduction of area from 1 to 14 percent over heats V-13 and V-15 (less than 0.0004 percent B and 0.01 percent Zr).

Boron plus zirconium additions.- Although recovered additions of 0.0069 to 0.0090 percent boron and 0.04 to 0.09 percent zirconium to heats V-18 and V-19 melted in alumina crucibles increased both rupture life and ductility, the increases were not so great as those experienced with 0.0089 percent boron and 0.01 percent zirconium (heat V-14). Figure 12 shows that, in the presence of 0.0069 to 0.0090 percent boron, there is apparently an optimum amount of zirconium around 0.01 weight percent. It should be noted that heats V-18 and V-19 had to be rolled from 2,000° F rather than from 2,150° F because of hot shortness. In addition, it is possible that use of the 2,300° F homogenizing treatment before rolling had an adverse effect when the boron and zirconium level was comparatively high. Therefore, any conclusions drawn from figure 12 should be qualified by recognizing that the differences in hot-working temperatures and the use of the 2,300° F treatment could have influenced properties.

Microstructures.- Examination was made of the rolled and heat-treated microstructures of boron- and zirconium-bearing heats (fig. 13). Heats V-18 (0.0069 percent B and 0.09 percent Zr) and V-19 (0.0090 percent B and 0.04 percent Zr) contained excess-phase stringers (fig. 13(d)) which were not found in other heats involved in this program. It is possible that the lower properties shown in figure 12 are associated with the presence of the stringers. Furthermore, the persistent internal rupturing of heats V-18 and V-19 during hot-rolling after recrystallization of the as-cast structure may be related to this phase.

Influence on creep characteristics.- In general, the addition of boron and/or zirconium did not alter primary creep significantly. Addition of boron seemed to lower the minimum second-stage creep rate. Both boron and zirconium did prolong third-stage creep greatly, mainly by increasing the deformation before fracture.

DISCUSSION

The properties of the experimental alloy were influenced to a marked extent by the type of ceramic used for induction melting in vacuum. The chemical analyses indicate that, if boron or zirconium were not introduced into the melt by reaction with the crucible, rather inferior properties resulted. There seems to be little doubt that the effects of the reaction with crucibles can be a major factor in the variation in strength and ductility at high temperatures and in hot-workability of vacuum-melted heat-resistant alloys bearing titanium and aluminum.

Most commercial induction vacuum melting uses magnesium or high-magnesium-content refractories for crucibles. Variation in the amount of reaction with the crucible could be and probably is a source of variable properties. Less effect in the second than in the first heat from a magnesia crucible suggests that the effect varies between successive heats from one crucible. The use of patching between heats and other possibilities of erratically providing fresh ceramic surfaces with increased reactivity to melts could be involved. Consideration of the possibility of other sources of boron compounds, such as ceramic binders, should be recognized.

The data indicate that boron derived from magnesia crucibles is the major cause of improvement in properties for heats made in magnesia. However, although such an effect was not substantiated by the chemical analyses, magnesium derived from the crucible could also be involved. So-called deoxidation with magnesium is commonly used to enhance the properties of high-nickel alloys. The improvements in properties obtained seem rather large for such small boron additions. However, heats made in alumina crucibles with boron additions had properties similar to those of heats made in magnesia with comparable boron levels. This suggests that magnesia crucibles do not contribute much through introduction of magnesium.

Boron compounds are usually associated with magnesia as a contaminant. Therefore, the derivation of boron from the crucible appears to be readily explainable. However, it is probable that recoveries of boron vary with a number of factors including the temperature and time of contact of the melt with the crucible, the ratio of crucible surface to melt volume, the number of heats, the variation in boron content of the magnesia crucible, and probably the compositional variations of the alloy.

The stability of the crucible materials may differ with melting atmosphere and pressure. Although considerable melt-crucible reaction occurred in vacuum melting, such crucible reactions may be limited in air melting by the higher pressure and the presence of slag and skull on the crucible walls.

The results also serve to suggest that the inadvertent introduction of even minute amounts of boron with the charge could be responsible for considerable variation in properties. The chances are that this occurs frequently, even though it may be unrecognized.

The data indicate that there are optimum amounts of boron when other trace elements, such as zirconium, are present. It seems probable that this has operated to give confusing results when such elements as boron and zirconium were knowingly or unknowingly added to heats simultaneously. There is a good chance that alloying elements other than boron and zirconium which are not yet recognized could exert a profound effect.

The relationship of the analyzed boron and zirconium contents to properties should not be accepted as complete establishment of compositional effects without further proof. There may be efficiency effects depending on their reaction with other elements not present in the heats studied so that total boron or zirconium content may not be directly related to properties. Furthermore, there is a possibility that heat and ingot size along with hot-working conditions were influencing results. It is also important to realize that analysis methods for boron and zirconium are subject to variation and that different laboratories might differ in the amounts reported.

The "homogenization" treatment at $2,300^{\circ}$ F for the ingots was introduced as the result of hot-working difficulties with early heats made in zirconia crucibles. Treatment of an ingot at $2,300^{\circ}$ F apparently improved hot-workability. It is now recognized that the improvement was probably due to an unusually high zirconium pickup from the crucible for the particular heat. It is possible, but by no means certain, that heating to $2,300^{\circ}$ F contributed to internal cracking of the high-boron or boron-plus-zirconium heats. Otherwise nothing has yet been found that suggests that the $2,300^{\circ}$ F treatment of the ingots influenced properties.

It is possible that the necessity for reducing the working temperatures of high-boron heats containing zirconium influenced the properties. Thus the fall off in strength with increasing zirconium content for boron-containing heats may be a reflection of the necessary change in hot-working conditions rather than a true compositional effect.

The rupture tests were carried out on material heat-treated at $2,150^{\circ}$ F and air-cooled. This differs from the usual heat-treatment of 4 hours at $1,975^{\circ}$ F plus 24 hours at $1,550^{\circ}$ F plus 16 hours at $1,400^{\circ}$ F. Other research in progress indicates that the treatment at $2,150^{\circ}$ F minimizes variation in properties due to the influence of hot-working conditions. In particular, it appears to prevent abnormally low strength sometimes encountered when a $1,975^{\circ}$ F treatment is applied to as-hot-worked material. A subsequent treatment at $1,975^{\circ}$ F after a $2,150^{\circ}$ F treatment usually gives the same properties as a single treatment at $2,150^{\circ}$ F. Very little difference in rupture strength and ductility has been observed for inclusion or omission of the aging treatments. Possibly the 4 hours used to equalize temperatures before testing cause the inclusion or omission of prior aging to have little influence. All experiences to date indicate the test results reported for a simple solution treatment at $2,150^{\circ}$ F to be generally characteristic of the alloy when treated at $1,975^{\circ}$ F except for the elimination of occasional low strength values.

Carbon content of the alloys varied more than was desired. The major reason for the variability of carbon content was the wide range of melting conditions employed with the consequent difficulty of controlling carbon recovery. This may be an additional factor in the variation of

properties. Insofar as could be ascertained, however, the only noticeable effect of the carbon content was the relatively high ductility in the rupture tests for those heats melted in zirconia crucibles with more than 0.12 percent of carbon. No correlation of carbon content with rupture life was apparent.

The data on the influence of titanium and aluminum content are not so complete as would be desirable. More variation than was intended occurred in the heats. As noted previously, there apparently was longer rupture life with a given zirconium content when titanium-plus-aluminum content was high. This probably resulted from the higher titanium-plus-aluminum level providing a larger percent by volume of $Ni_3(Al, Ti)$ during stress-rupture testing.

The relationships between crucible ceramics and properties were developed for induction vacuum melting. The amount of such effects under other conditions of melting is not clear at this point. It is interesting to note that the ductility in rupture tests was low when the alloy was made in alumina crucibles. Low-zirconium heats made in zirconia crucibles also had low ductility. This suggests that a part of the usual increase in ductility attributed to vacuum melting may be due to the opportunity offered by vacuum melting to introduce effective trace elements from crucibles. In addition, addition of trace elements other than boron and zirconium by other melting procedures may alter the effectiveness of a given boron and zirconium level.

The crucible-reaction effects were discovered as a result of inability to define the effects of variable-melting practices and the role of oxygen and nitrogen, the original objectives of the investigation. The crucible reactions must be controlled before the influence of the melting variables can be defined. Because of the sensitivity of properties to reactions with magnesia along with interrelated effects from such factors as hot-workability, very careful research will be necessary to define the effects clearly. While the data of this report suggest that many melting variables are secondary to the introduction of boron or zirconium from the crucibles, there may be extenuating circumstances which mask the effects. The subject is of sufficient complexity that probably the only way it can be cleared up rests in the clarification of the mechanism by which as little as 15 parts per million of boron can increase rupture life 5 times.

In spite of the limitations on the generality of the results it is believed that the data reported reflect production experience quite well. Many of the variations in properties in the experimental investigation and in commercially produced alloys of the type investigated seem to be explainable in terms of the results of the investigation.

CONCLUSIONS

The study of the influence of crucible materials on the high-temperature properties of a vacuum-melted nickel-chromium-cobalt alloy resulted in the following results and conclusions:

1. The type of ceramic crucible used in the melting was an important variable in induction vacuum melting of the experimental alloy. A review of previous experience indicates that this explains the variability of properties of many vacuum-melted alloys.
2. Heats of the experimental 55Ni-20Cr-15Co-4Mo-3Ti-3Al alloy melted in alumina crucibles exhibited comparatively short rupture life and low ductility with a high susceptibility to cracking.
3. Rupture life and ductility and resistance to cracking during hot-working were benefited by melting in zirconia crucibles. The benefits were related to the zirconium contents derived from reaction of the melts with the crucibles.
4. Pronounced benefits to rupture life and ductility and resistance to cracking during hot-rolling resulted from melting in magnesia crucibles. In this case, the benefits appeared to be due to boron derived from crucible materials.
5. Deliberate addition of boron or zirconium to heats melted in alumina crucibles resulted in properties similar to those obtained by derivation of these elements from the zirconia or magnesia crucibles.
6. Simultaneous additions of boron and zirconium in optimum amounts led to higher creep-rupture properties than did additions of either element alone. The data also indicate that there are optimum amounts of boron-plus-zirconium content beyond which rupture properties fall off and susceptibility to cracking during hot-working increases.
7. The results reported serve to clarify many puzzling and seemingly contradictory variations in properties in both experimental and commercial heats. In particular, variable boron pickup from the usual magnesia refractories used in commercial vacuum melting could explain many of the variations. Unrecognized introduction of boron or zirconium with melting stock could be another source of variability. Interrelationships between boron and zirconium content leading to low strength beyond certain amounts are probably a source of misunderstood low strengths when such elements have been purposely added.

University of Michigan,
Ann Arbor, Mich., January 18, 1957.

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TABLE I
MELTING PRACTICE FOR EXPERIMENTAL HEATS

Heat	Crucible	Heat in crucible	Refining time, min	Refining temperature, °F	Decoxidant	Superheat temperature, °F	Pouring temperature, °F	Addition, percent
V-1	Zirconia	1	0	-----	C in charge	3,050	3,000	-----
V-2	Zirconia	2	0	-----	C in charge	3,050	3,000	-----
V-3	Zirconia	1	20	2,700	C in charge plus chunk C	3,000	3,000	-----
V-4	Zirconia	1	20	2,700	Chunk C	2,900	2,900	-----
^a V-5	Zirconia	2	20	2,700	Chunk C	2,950	2,950	-----
V-6	Zirconia	3	20	2,700	Al	3,100	3,000	-----
V-7	Zirconia	4	20	2,700	Si	2,975	2,975	-----
V-8	Zirconia	1	20	2,700	Al	2,975	2,750	-----
V-9	Zirconia	2	20	2,700	Al	2,750	2,750	-----
V-10	Zirconia	3	20	2,900	Al	3,100	3,000	-----
V-11	Magnesia	1	0	-----	C in charge	3,000	3,000	-----
V-12	Magnesia	2	0	-----	C in charge	3,000	3,000	0.01B
V-13	Alumina	1	0	-----	C in charge	3,000	3,000	-----
V-14	Zirconia	1	0	-----	C in charge	3,000	3,000	.01B
V-15	Alumina	1	0	-----	C in charge	3,000	3,000	-----
V-16	Alumina	2	0	-----	C in charge	3,000	3,000	.15Zr
V-17	Alumina	3	0	-----	C in charge	3,000	3,000	.01B
V-18	Alumina	4	0	-----	C in charge	3,000	3,000	.01B, 0.10Zr
V-19	Alumina	5	0	-----	C in charge	3,000	3,000	.01B, 0.10Zr
V-20	Magnesia	1	0	-----	C in charge	3,000	3,000	-----
^b V-21	Magnesia	2	0	-----	C in charge	3,000	3,000	-----
V-24	Alumina	6	0	-----	C in charge	3,000	3,000	.0025B

^aCast in tapered mold (other heats cast in straight mold).

^bMelt-down time, 2 hr (other heats, 1 hr).

TABLE II
CRUCIBLE MATERIALS

Material	Brand name	Supplier	Chemical composition, weight percent								
			Al ₂ O ₃	ZrO ₂	MgO	SiO ₂	CaO	Fe ₂ O ₃	TiO ₂	B ₂ O ₃	Alkalies
Alumina	Taycor	The Charles Taylor's Sons Co.	87.6	----	0.04	11.4	0.03	0.21	0.07	Not detected	0.41
Zirconia	Stabilized zirconia	Titanium Alloy Mfg. Div., National Lead Co.	.02	93.8	.05	.5	4.9	.02	.63	Not detected	<.01
Magnesia	Magnorite	Norton Co.	-----	-----	97.0	1.5 to 2.0	1.3 to 1.5	0.1 to 0.2	----	0.1 to 0.2	----

TABLE III
RESULTS OF CHEMICAL ANALYSIS OF HEATS
[Balance is nickel]

Heat	Elements present, weight percent															
	Cr	Co	Mo	Ti	Al	Si	Mn	C	S	P	Zr	B	Ca	Mg	Fe	Cu
V-1	21.2	14.9	4.00	2.82	3.30	0.12	0.13	0.05	----	0.003	0.09	-----	----	<0.01	<0.20	<0.10
V-2	21.7	14.6	4.00	3.17	3.45	.14	<.10	.04	----	.004	.08	-----	----	<.01	<.20	<.10
V-3	20.7	14.8	4.00	3.22	3.35	.11	.16	.06	----	.005	.04	0.0004	----	<.01	<.20	<.10
V-4	18.1	15.2	4.10	2.98	3.10	.10	.10	.05	----	.006	.06	.0005	----	<.01	<.30	<.10
V-5	18.2	15.1	4.20	3.18	3.15	.10	.10	.09	----	.008	.09	-----	----	<.01	<.30	<.10
V-6	18.8	15.1	4.15	3.14	3.14	.10	<.10	.08	0.008	.003	.19	.0004	----	-----	<.30	-----
V-7	19.4	15.0	4.10	3.30	3.35	.14	.15	.08	----	.006	<.03	-----	----	-----	<.30	-----
V-8	19.2	14.5	4.20	2.98	3.00	.22	.15	.20	----	.007	<.03	-----	----	<.01	-----	<.10
V-9	19.8	14.5	4.10	2.98	2.85	.23	.13	.19	----	.004	<.03	.0004	----	<.01	-----	<.10
V-10	19.8	15.2	4.10	3.05	3.15	.12	.10	.13	----	.005	.06	-----	----	<.01	<.30	<.10
V-11	20.0	16.2	4.20	3.25	3.58	.18	.10	.05	----	.004	<.01	.0017	----	<.01	<.30	<.10
V-12	20.9	14.8	4.20	3.15	3.25	.20	<.10	.10	----	.004	<.01	.0089	----	<.01	<.30	<.10
V-13	20.4	14.8	4.20	3.25	3.57	.25	.12	.05	----	.007	<.01	.0003	<0.01	<.001	<.01	<.01
V-14	20.8	14.8	4.20	3.20	3.30	.19	.11	.09	----	.007	.01	.0088	----	<.01	<.30	<.10
V-15	19.7	15.0	3.90	3.08	3.35	.17	.13	.08	.007	.006	<.01	.0002	----	<.01	<.30	<.10
V-16	20.0	16.0	3.90	3.12	3.39	.17	.10	.06	----	.006	.13	.0009	----	<.01	<.30	<.10
V-17	19.4	16.5	3.75	3.02	3.26	.16	.12	.07	----	.008	<.01	.0074	----	<.01	<.30	<.10
V-18	19.8	15.7	3.70	3.10	3.26	.12	.13	.07	----	.006	.09	.0069	----	<.01	<.30	<.10
V-19	20.8	16.0	3.90	3.17	3.58	.13	.12	.08	----	.006	.04	.0090	----	<.01	<.30	<.10
V-20	-----	-----	-----	-----	-----	-----	-----	.12	----	.007	-----	.0010	<.01	<.001	<.01	<.01
V-21	-----	-----	-----	-----	-----	-----	-----	.15	----	.007	-----	.0004	<.01	<.001	<.01	<.01
V-24	19.8	15.9	4.05	2.92	3.06	.16	<.10	.13	----	-----	<.01	.0012	<.10	<.01	<.30	-----

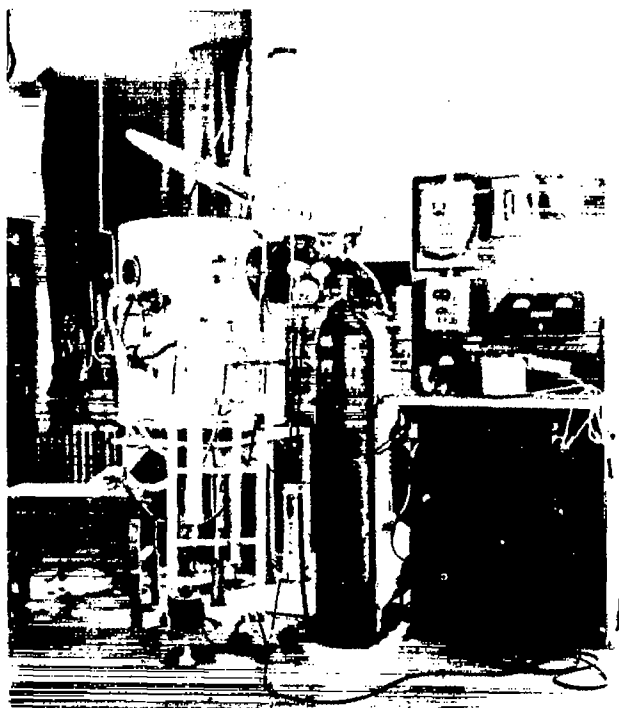
TABLE IV

STRESS-RUPTURE DATA AT 1,600° F AND 25,000 PSI

[As-rolled bar stock was heat-treated 2 hours at 2,150° F and air-cooled unless otherwise noted; specimens were preheated 4 hours at test temperature before loading]

Heat	Crucible material	Rupture time, hr	Elongation, percent	Reduction of area, percent	Compositional variable, percent			
					C	Zr	B	Ti + Al
V-1	Zirconia	^a 82.3 96.6	-- 5	5 6	0.05	0.09	-----	6.12
V-2	Zirconia	117.7 143.5	8 5	3 6	.04	.08	-----	6.62
V-3	Zirconia	90.7 126.0	4 5	4 4	.06	.04	0.0004	6.57
V-4	Zirconia	^a 99.5 106.3	6 4	4 5	.05	.06	.0005	6.08
V-5	Zirconia	^a 87.1 91.2	6 5	6 6	.09	.09	-----	6.33
V-6	Zirconia	147.4 133.7	5 6	5 8	.08	.19	.0004	6.28
V-7	Zirconia	75.5 103.1	4 5	3 5	.08	<.03	-----	6.65
V-8	Zirconia	81.2 66.3	8 11	12 9	.20	<.03	-----	5.98
V-9	Zirconia	50.8 54.6	8 6	12 8	.19	<.03	.0004	5.78
V-10	Zirconia	83.5 86.5 79.9 102.8	9 8 6 8	9 10 6 9	.13	.06	-----	6.20
V-11	Magnesia	185.2 229.9	3 5	2 5	.05	<.01	.0017	6.83
V-12	Magnesia	428.8 393.5	10 7	11 8	.10	<.01	.0089	6.40
V-13	Alumina	56.9 47.5	2 1	2 1	.05	<.01	.0003	6.62
V-14	Zirconia	666.3 626.8	17 12	16 13	.09	.01	.0088	6.50
V-15	Alumina	44.6 52.0	2 2	1 1	.08	<.01	.0002	6.43
V-16	Alumina	144.0 118.9	4 4	4 4	.06	.13	.0009	6.51
V-17	Alumina	400.4 346.0	7 7	6 5	.07	<.01	.0074	6.28
V-18	Alumina	243.8 304.5	7 10	6 10	.07	.09	.0069	6.36
V-19	Alumina	546.6	11	12	.08	.04	.0090	6.55
V-20	Magnesia, first heat	223.2 206.9	5 4	4 3	.12	<.01	.0010	----
V-21	Magnesia, second heat	103.4 106.0	4 3	2 2	.15	<.01	.0004	----
V-24	Alumina	170.0	5	3	----	<.01	.0012	5.98

^aHeat-treated 1 hr at 2,150° F, air-cooled.



(a) External view.



(b) Internal view of crucible, charge buckets, and ingot mold.

Figure 1.- University of Michigan vacuum-melting furnace.

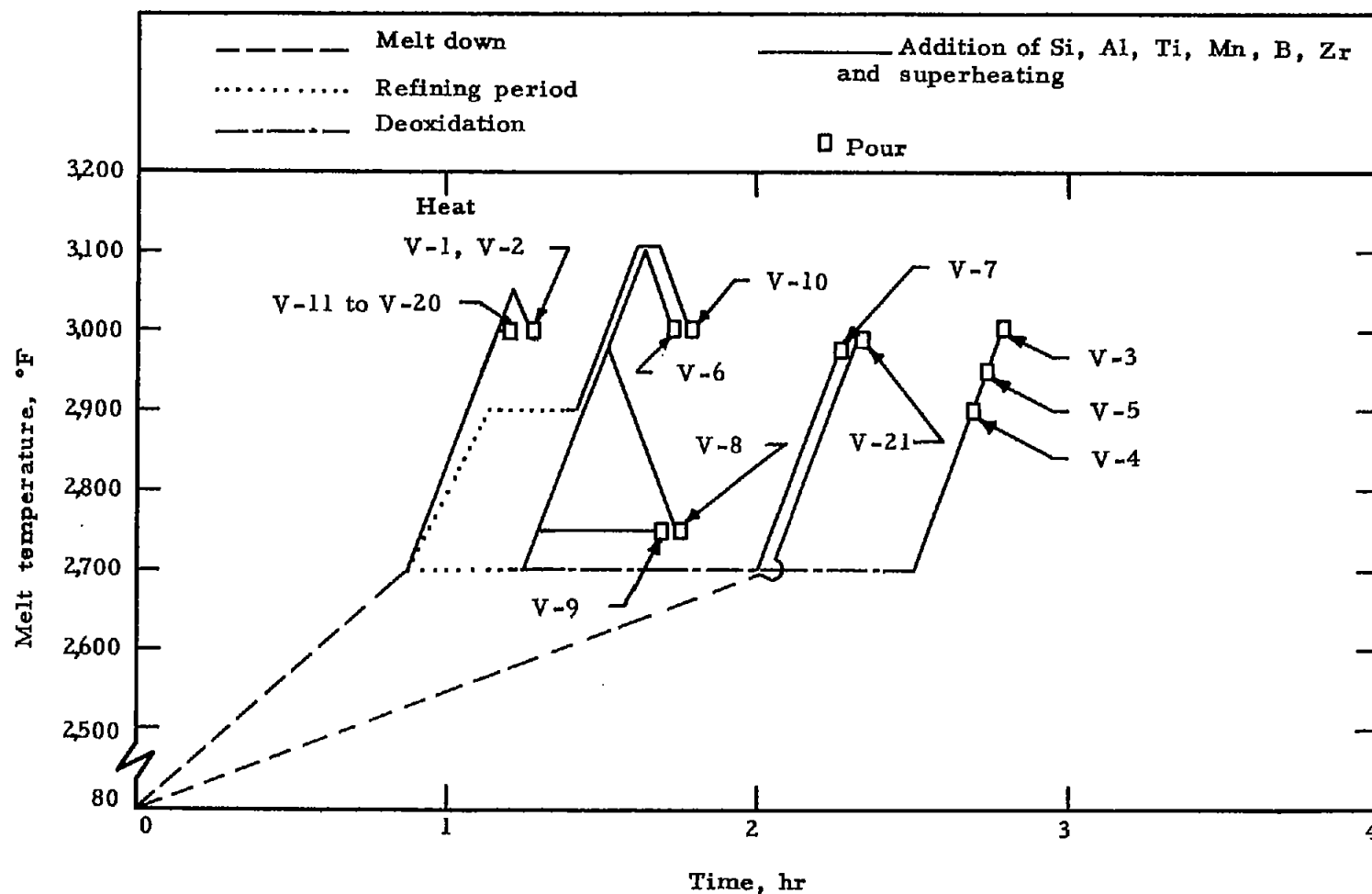
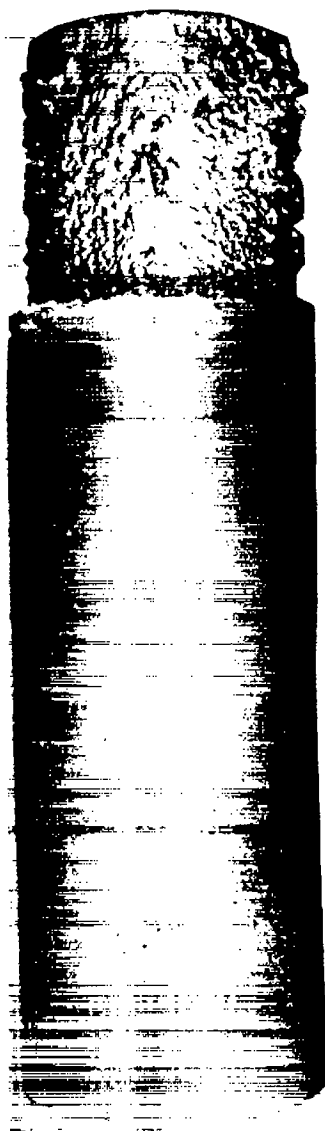


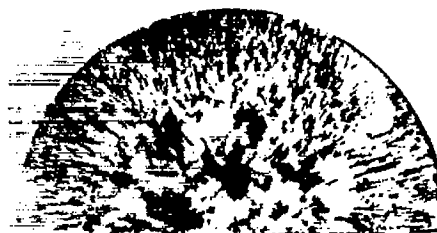
Figure 2.- Melting cycles used in study of effect of melting variables on high-temperature properties of experimental alloy.



(a) As-cast ingot 9 inches long with 2.5-inch diameter; weight, 10 pounds.

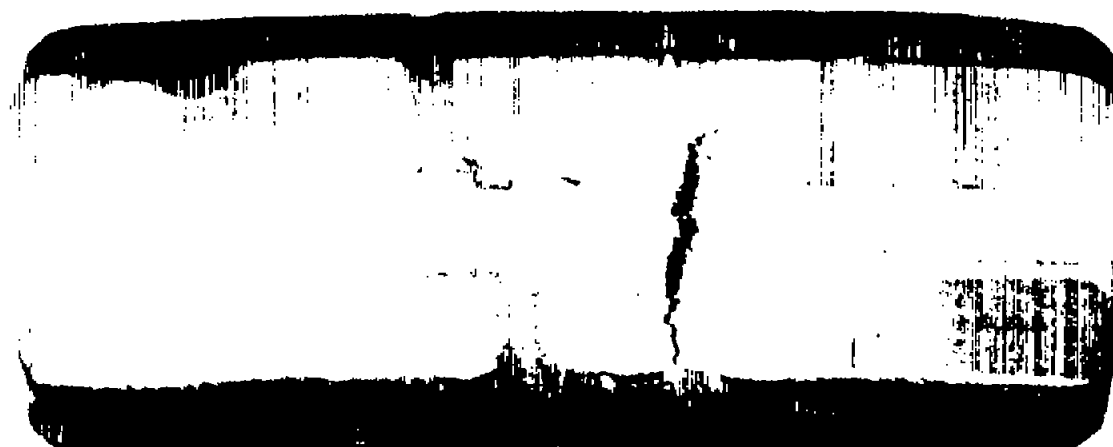


(b) Section of hot top showing isolation of pipe to hot top.

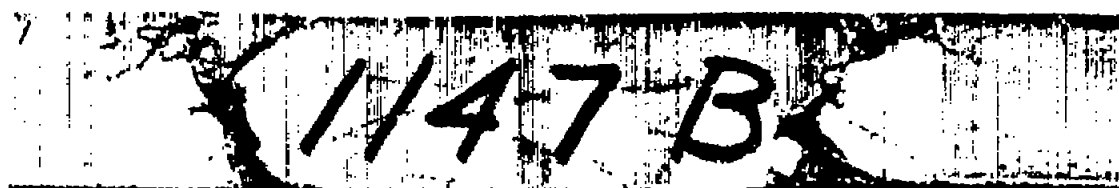


(c) Macrostructure of ingot section.

Figure 3.- Typical as-cast ingot for experimental heats.

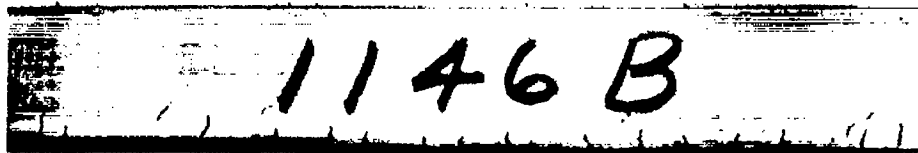


(a) Ingot after three passes at 2,150° F.



(b) Bar stock after reduction to 7/8-inch square. Large cracks occurred during ingot breakdown and could not be ground out.

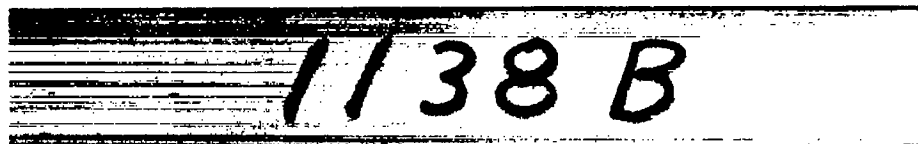
Figure 4.- Internal rupture obtained during ingot breakdown of heat V-14 with 0.0088 percent boron and 0.01 percent zirconium and resultant bar stock after continued hot-rolling.



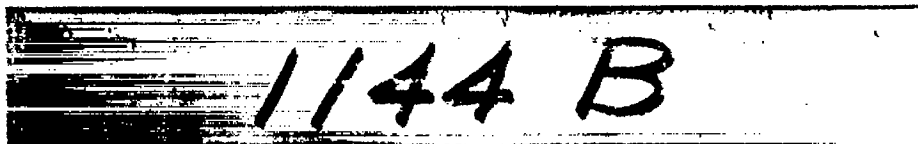
(a) Heat V-13 with 0.0003 percent boron and less than 0.01 percent zirconium.



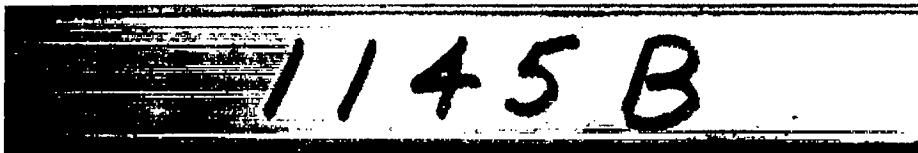
(b) Heat V-1 with 0.09 percent zirconium melted in zirconia with no boron addition.



(c) Heat V-6 with 0.19 percent zirconium and 0.0004 percent boron.



(d) Heat V-11 with 0.0017 percent boron and less than 0.01 percent zirconium.



(e) Heat V-12 with 0.0089 percent boron and less than 0.01 percent zirconium.

Figure 5.- Effect of boron or zirconium content on corner cracking of experimental heats when rolled to 7/8-inch-square bars at 2,150° F.

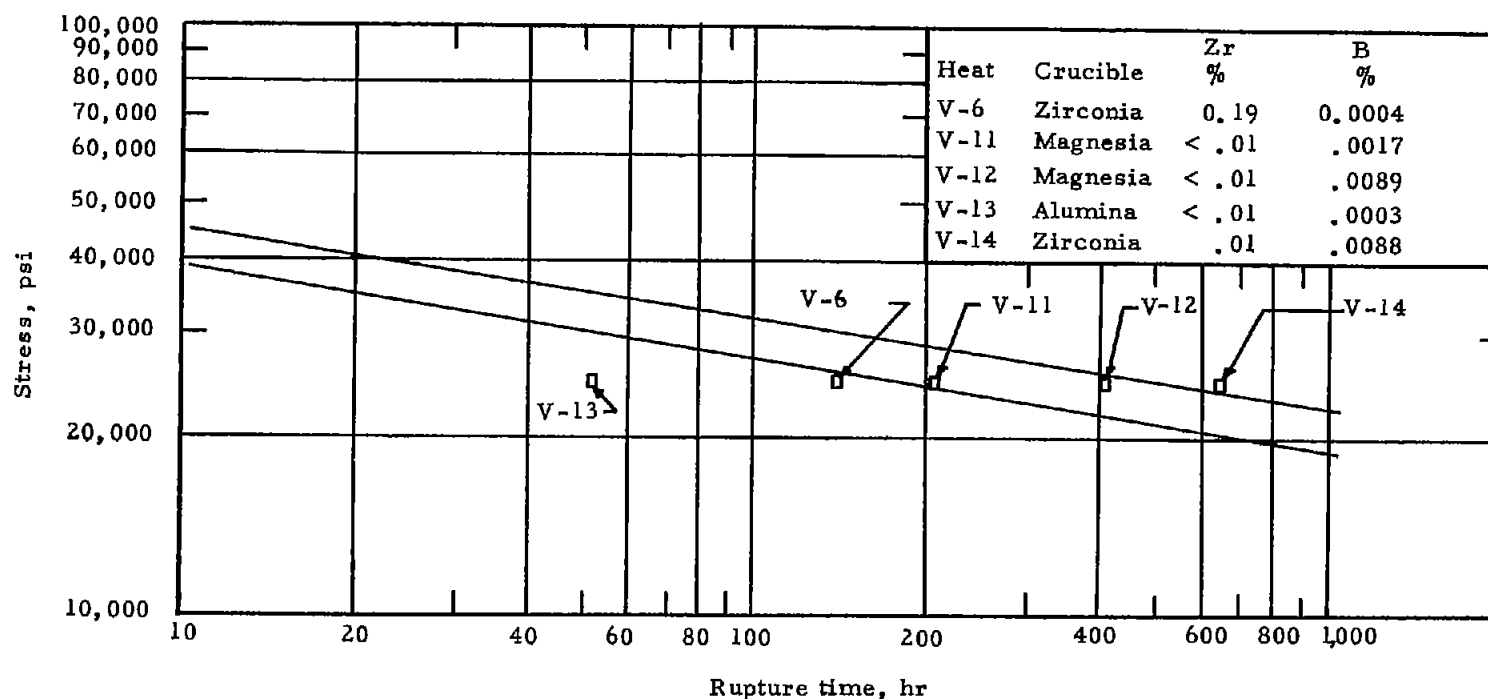


Figure 6.- Average rupture life of experimental heats plotted on rupture band at 1,600° F for two heats of Ulmet 500 as reported by Utica Drop Forge and Tool Corp. Treatment of experimental heats was 2 hours at 2,150° F, then air-cooled. Treatment of Utica heats was 2 hours at 2,150° F, air-cooled plus 4 hours at 1,975° F, and air-cooled plus 24 hours at 1,550° F plus 16 hours at 1,400° F.

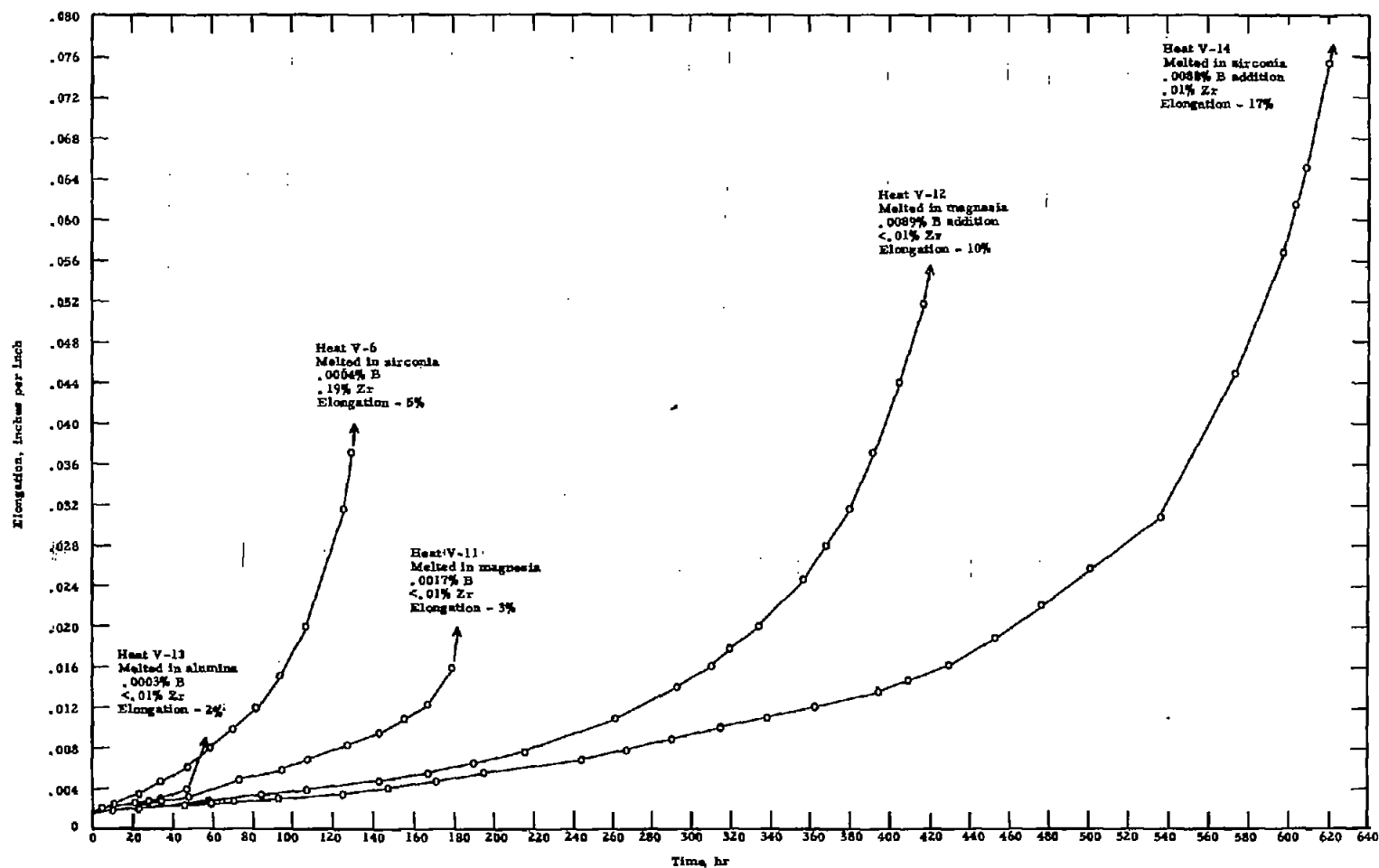


Figure 7.- Comparative creep curves at 1,600° F and 25,000 psi for experimental heats showing effect of boron and zirconium contents on creep characteristics.

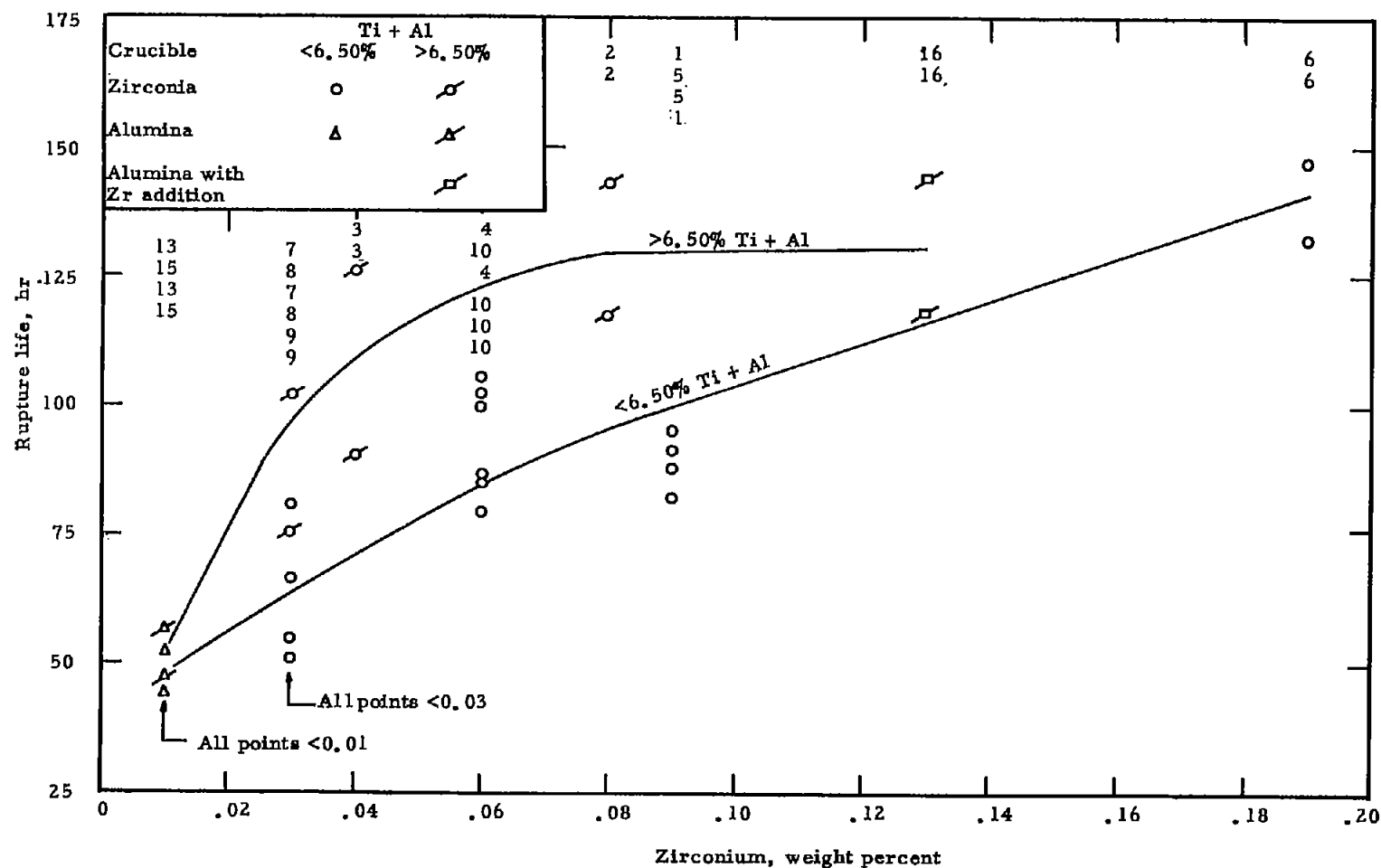


Figure 8.- Effect of zirconium content on rupture life at 1,600° F and 25,000 psi of experimental heats melted in zirconia or alumina (boron not influential). Heat-treatment was 2 hours at 2,150° F, then air-cooled. Numbers at top of figure identify V- heat number for data points directly below numbers.

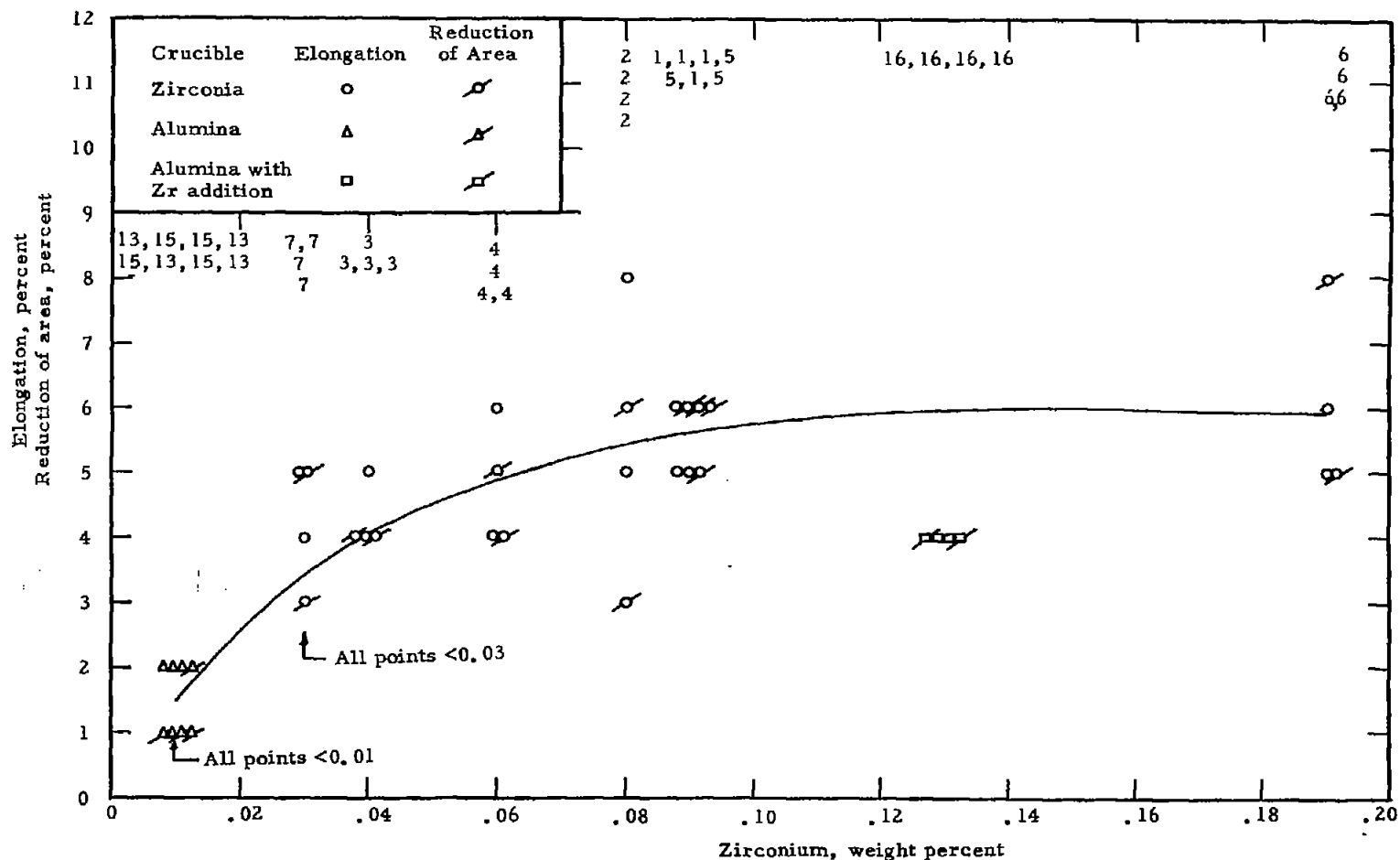


Figure 9.- Effect of zirconium content on ductility at 1,600° F and 25,000 psi of experimental heats melted in zirconia or alumina (boron not influential). Heats have carbon content of 0.04 to 0.09 percent. Heat-treatment was 2 hours at 2,150° F, then air-cooled. Numbers at top of figure identify V- heat number for data points directly below numbers.

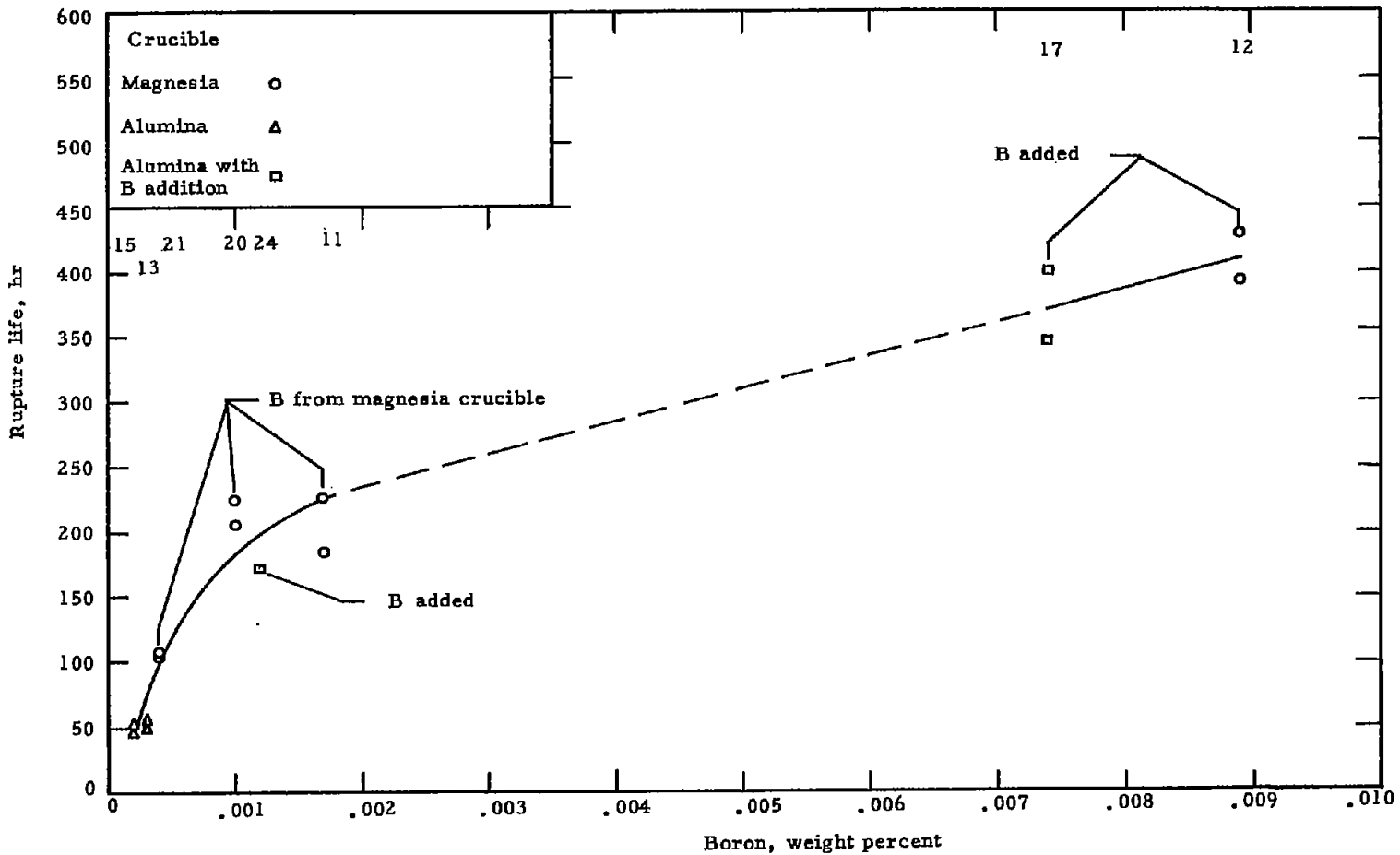


Figure 10.- Effect of boron content on rupture life at 1,600° F and 25,000 psi of experimental heats melted in alumina or magnesia (with less than 0.01 percent zirconium). Heat-treatment was 2 hours at 2,150° F, then air-cooled. Numbers at top of figure identify V- heat number for data points directly below each number.

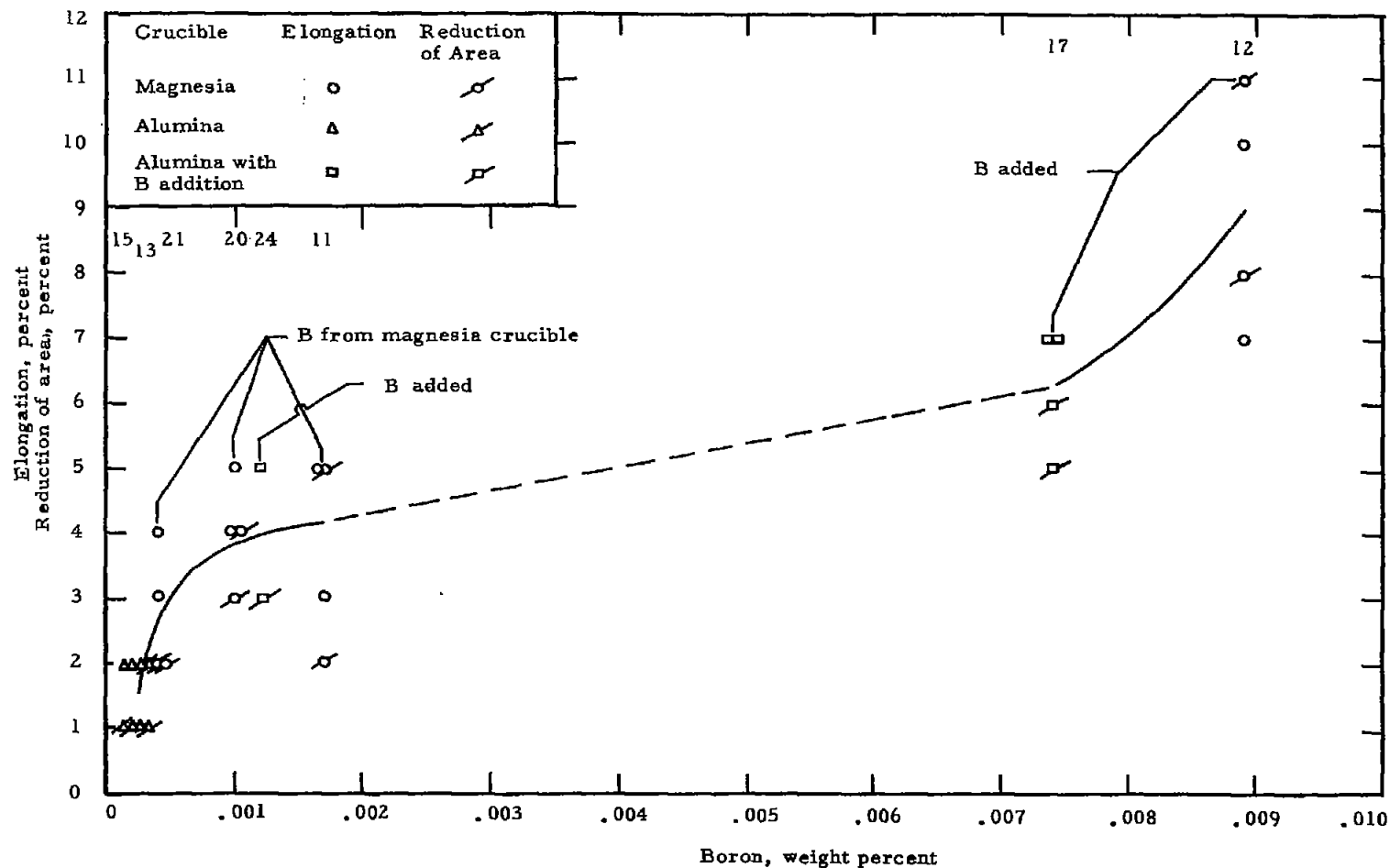


Figure 11.- Effect of boron content on ductility at 1,600° F and 25,000 psi of experimental heats melted in alumina or magnesia crucibles (with less than 0.01 percent Zr). Heat-treatment was 2 hours at 2,150° F, then air-cooled. Numbers at top of figure identify V-heat number for data points directly below each number.

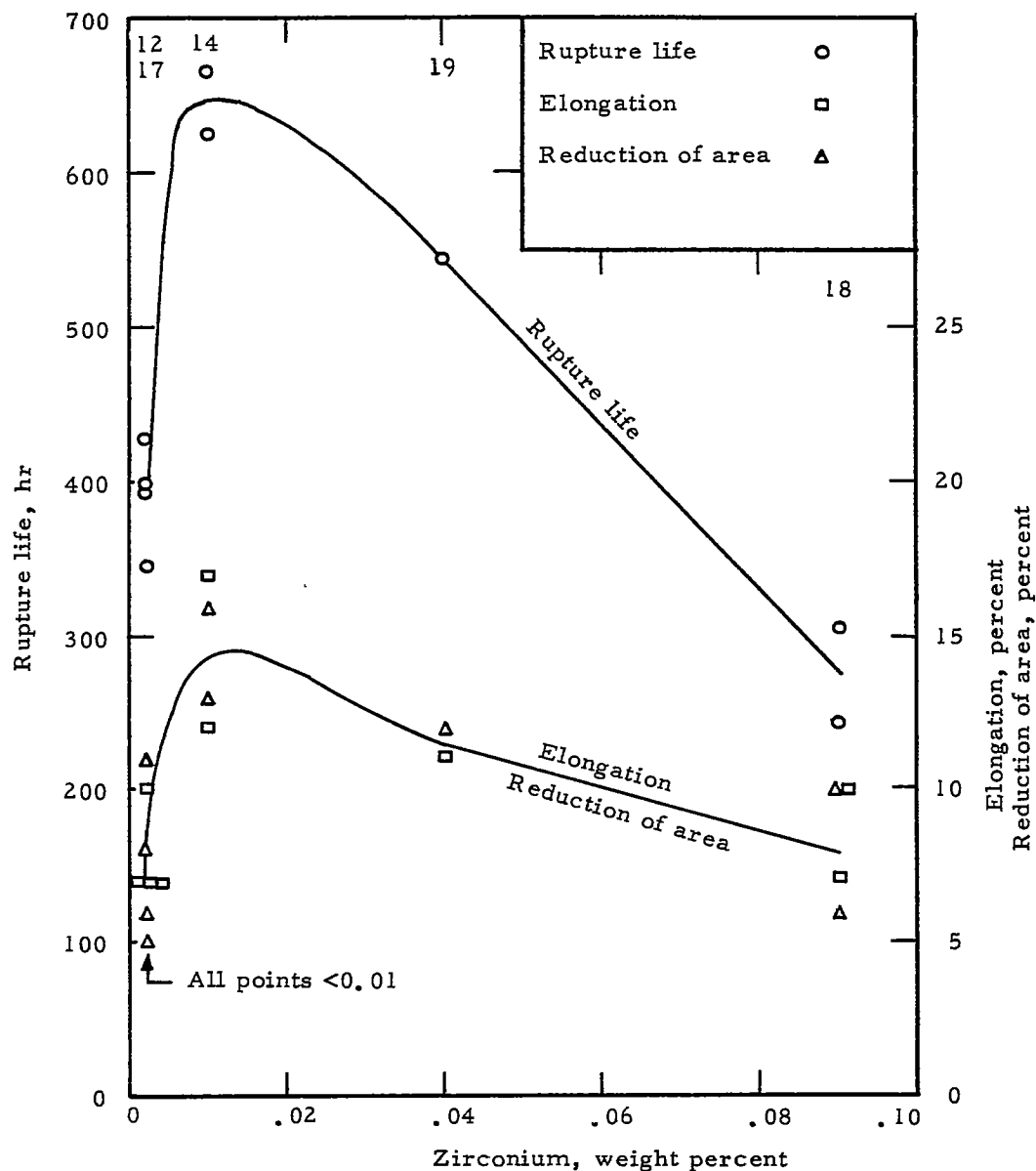
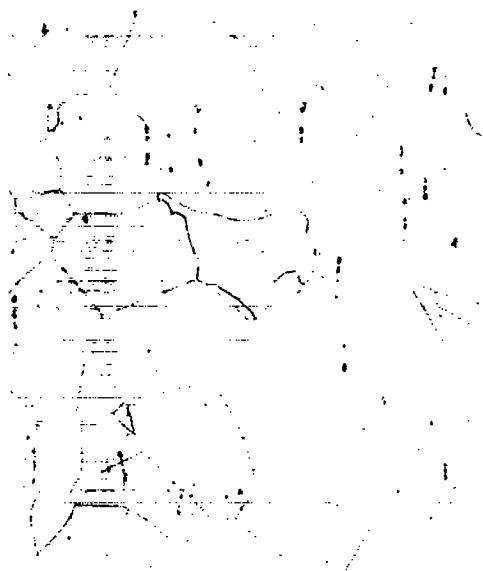
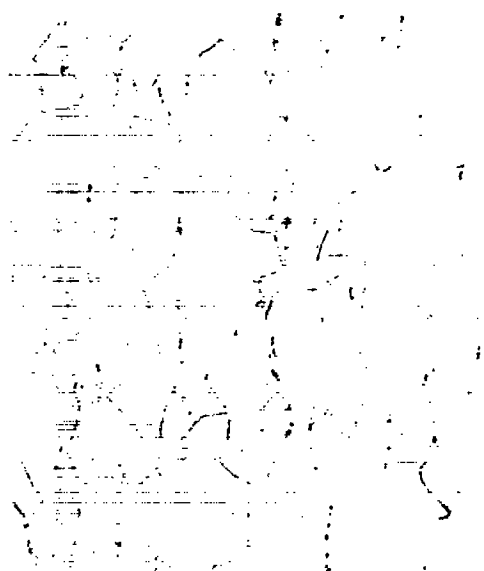


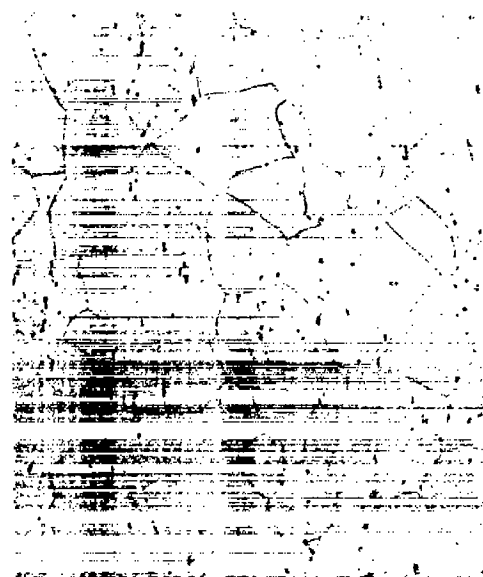
Figure 12.- Effect of zirconium content on rupture life and ductility of experimental heats at 1,600° F and 25,000 psi in presence of boron contents of 0.0069 to 0.0090 percent. Heat-treatment was 2 hours at 2,150° F, then air-cooled. All heats were homogenized 1 hour at 2,300° F before rolling. The heats with less than 0.03 percent zirconium were hot-rolled at 2,150° F while the heats with more than 0.03 percent zirconium were rolled at 2,000° F. Numbers at top of figure identify V- heat number for data points directly below each number.



(a) Heat V-13 with 0.0003 percent boron and less than 0.01 percent zirconium.



(b) Heat V-12 with 0.0089 percent boron and less than 0.01 percent zirconium.



(c) Heat V-14 with 0.0088 percent boron and 0.01 percent zirconium.



(d) Heat V-18 with 0.0069 percent boron and 0.09 percent zirconium.

Figure 13.- Effect of boron and zirconium on the microstructure of experimental heats. Specimens were heat-treated 2 hours at 2,150° F, then air-cooled, after rolling. Magnification, X100.